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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of Van De Mark, et al.
Serial No. 09/532,839
Filed March 21, 2000
For WATER BORNE FILM-FORMING COMPOSITIONS
Examiner Paul R. Michl

Art Unit 1755

March 22, 2002

DECLARATION OF PRIOR INVENTION UNDER 37 C.F.R. § 1.131

I, Michael R. Van De Mark, declare as follows:

1. I am a co-inventor of the subject matter claimed in the above-entitled United States patent application, Serial Number 09/532,839. Since January 1, 1986, I have been an Associate Professor of Chemistry at the University of Missouri Rolla

2. Nantana Jiratumnukul, a former graduate student of the University of Missouri Rolla, and I are named as co-inventors of the invention claimed in the above-identified patent application. We conceived and reduced to practice the invention claimed in this application in the United States before July, 1996.

3. All work referred to herein was carried out in the United States.

4. Evidence of our conception and reduction to practice appears in Exhibit A which is attached to this declaration. On information and belief, Exhibit A is a true and correct copy of two pages of a laboratory notebook maintained by Nantana Jiratumnukul who, at the time, was working under my direction and supervision. The work described on these two pages was carried out prior to July, 1996. Although the dates which appear on the originals of each these two pages were covered when the attached photocopies were made; each page bears a date prior to July, 1996. The first

page of Exhibit A (which bears page number 151) identifies a master batch for a paint formulation which contains PG (propylene glycol), water, surfactant, resin and a pH modifier which were mixed together before a coalescent aid was added. The stated objective was to compare "Cpd X" against the benchmark coalescent aid, Texanol. "Cpd X" was identified on the page to be ethylene glycol soyoil derivative. The second page of Exhibit A (which bears page number 149) describes the preparation of two paint formulations, each of which contained "Cpd X," i.e., ethylene glycol soyoil derivative. The other components of these paint formulations are as follows: water, PG (propylene glycol), triton X-100 (a surfactant), triton X-102 (a surfactant), AMP-95 (a pH modifier), Rhoplex 1018 (a resin), Drewplus 493 (a defoamer), ethylene glycol monobutyl ether. As such, Exhibit A evidences our conception and reduction to practice of the invention claimed in the application prior to July, 1996.

5. I was not aware of Bumanlag U.S. Patent No. 5,753,742, Rauls US Patent No. 6,156,833, or Saam U.S. Patent No. 6,177,510 prior to the filing of this application.

6. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Michael R. Van De Mark

March 22, 2002
Date

Master Batch

2/15/96

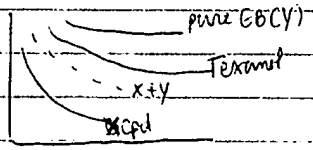
pe
H_2O
surfactant
resin
pH modifier
} mix together as master [illegible] then weigh and add coalescer and

Objective compare our cpd w/ benchmark = Texanol

Texanol	cpd X (our cpd)	EB (ethylene glycol monobutyl ether) (CY)
10%	8%	15
9%	6%	10
12%	4%	5

cpd X (our cpd) is Ethylene glycol soyoil derivative

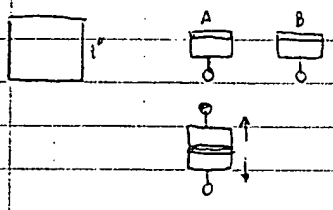
MFFT PLOT



1st year get MFFT plot and optimum condition

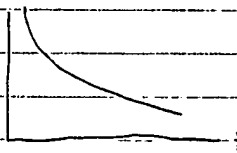
2nd year search for new test measuring : gloss, adhesion, impact resistance, hardness

Testing Block Resistance (using Instron)



1) paint surface w/ paint, leave them at different time interval
2) put A+B together, put under mass for 10 min
3) remove mass
4) test instron, see how much force need to apply to pull A+B

The longer time we wait, should be; dryer → not stick together, need less force



UCAR 379G polyvinyl acrylic
Flexbond 325 polyvinyl acetate

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2/18/96 Formulation

	lbs
H_2O	289
PG	43.6
triX-100	2.0
AMP-95	4.0
Rhoplex 1018	434.2
Drewplus 483	2.5
cpd x	5.0
butyl cellusolve	5.0
H_2O	100.18

replace the texanol 10.0

Result from drawn-down.

Texanol 1% gave a clear good film.

Compound x 1% gave hazy and cratering. This might be because cpd x was put too much. To solve the problem, amount of cpd x was cut and make up w/ butyl cellusolve of 50:50 ratio

butyl cellusolve (ethylene glycol monobutyl ether)

$C_4H_9-O-CH_2-CH_2-OH$ (increase hydrophilic

After mixing all the components left the paint overnight before drawing (w/ 3 mil thick film). The results from drawn-down was that there were some cratering and the surface of paint was not wet on the kanetta paper.

After the formulation was left for 2 days, the drawn-down had been taken again. There were remain some cratering although this time it was better than before. The drawing problem might be the surfactant used in formula. So, the wetting agent Zonyl APC from Du Pont was added about 5 drops. After mixing Zonyl APC into the formulation, it the cratering problem alot. Since Zonyl is very expensive. Thus we try to find other proper surfactant which has higher or lower HLB than triton x-102 to replace. compare the results so that we can predict the trend and choose the right type of surfactant to improve wetting.

triton x-100 octyl phenyl polyethoxy ethanol HLB = 13.5

triton x-102 " " = 14.6

* Agzepol CA-720 " " = 14.6 from Phone-Pac

2/20/96 Trial with the higher HLB surfactant [Triton x-102, HLB = 14.5]

	lbs
H_2O	289
PG	43.6
triton x-102	2.0
AMP95	4.0
Ucar 039	434.2
Drewplus	2.5
* Cpd-X	10.0
H_2O	100.8

* would be replaced by cpd x + EB, EB, texanol

PAINT/COATINGS DICTIONARY

Compiled by
Definitions Committee
Of the Federation of Societies for Coatings Technology

Stanley LeSota, *Chairman*

David J. Engler
Ruth Johnston-Feller
J. Richard Kiefer, Jr.
Seymour Mark
John Landis
Michael L. McGroarty
Lothar Sander
Francis J. Willard

Published by
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The Paint/Coatings Dictionary has been prepared through the dedicated efforts of many volunteers and was assembled by the staff members of the Federation of Societies for Coatings Technology. The information has been compiled from sources and by persons believed to be reliable; however, the Federation does not assume any responsibility for the use or misuse of the information contained in this dictionary nor guarantee its accuracy or completeness.

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Clouding Development in a clear varnish or lacquer film or liquid of an opalescence or cloudiness caused by the precipitation of insoluble matter or immiscibility of components.[161](BSI)

Cloud Point Point at which a definite lack of clarity (cloudiness) appears when a liquid is subject to adulteration or when it is mixed with another substance, or the temperature at which a liquid becomes cloudy when it is cooled.[157,161]

Clupanodonic Acid Acid found in many fish oils. The acid possesses an unusually high iodine value—well in excess of 300.[63] *Also called Docosapentanoic Acid.*

CMC *See Carboxymethyl Cellulose.*[83,139,155]

Coacervate An aggregate of colloidal droplets held together by electrostatic attractive forces.[139]

Coacervation Reversible collection of emulsoid particles into liquid droplets preceding flocculation. An intermediate stage between sol and gel formation.[139] *cf. Coagulation.*

Coagulation (1) Process whereby a fluid liquid is changed into a thickened, curdled or congealed mass.[42,131,161] (2) Irreversible agglomeration of particles originally dispersed in a rubber latex.[42,131,161](ASTM)

Coagulum An agglomerate of particles.[42,161,139](ASTM)

Coalescence The formation of a film of resinous or polymeric material when water evaporates from an emulsion or latex system, permitting contact and fusion of adjacent latex particles.[55](DAC) Action of the joining of particles into a film as the volatile evaporates.[55]

Coalescent (Coalescing Agent) Solvent with a high bp which, when added to a coating, aids in film formation via temporary plasticization (softening) of the vehicle.[55,83,164]

Coal Tar A dark brown to black cementitious material produced by the destructive distillation of bituminous coal.[76](ASTM)

Coal Tar Colors *See Aniline Pigments.*[41]

Coal Tar-epoxy Coating Coating in which binder or vehicle is a combination of coal tar with epoxy resin.[71]

Coal Tar Hydrocarbons Aromatic hydrocarbons derived from coal tar, including benzene, toluene, xylene, naphtha, etc.[164]

Coal Tar Pitch Distillation residue from coal tar. It varies considerably from a very soft to a very hard product. Fusion points vary from as low as 27°C (80°F) to as high as 232°C (450°F).[76]